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## SMOKING COMPOSITION SHEET

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Claim

A smoking composition sheet, characterized by compounding a reconstituted tobacco raw material sheet containing carboxymethylcellulose as a binder impregnated with a volatile flavoring agent by adsorption to form a slurry which is subsequently molded and dried to enhance the volatile flavoring agent-retaining ability.

Detailed explanation of the inventionIndustrial application field

This invention pertains to a smoking composition. In particular, it pertains to a smoking composition sheet with its ability to retain a flavoring agent improved by allowing a powder or granular adsorbent to adsorb a volatile flavoring agent and using the flavoring agent-adsorbed material as a part of the raw materials of the reconstituted tobacco sheet.

Prior art and its problems

Various kinds of flavoring agents are generally added to the raw material tobacco leaves, cuts or intermediate cuts during a process for the production of tobacco, thus allowing respective brands to have their unique flavors and aromas. Some of those flavoring agents used in this case are strongly volatile and evaporate quickly during processing, reducing the effects of adding the flavoring agent. In addition, the higher the temperature, the more volatile are these volatile flavoring agents, and consequently, the flavor and aroma are changed between immediately after lighting and near the end of burning. The combustion temperature is especially high in the case of smoking pipe tobacco; thus, the taste of the flavoring agent added is strong and concentrated at the beginning of smoking, whereas they tend to become weak and faint as it approaches the end. For example, menthol is strongly volatile, and the tendencies described above are very remarkable. Therefore, it is necessary to retain those strongly volatile flavoring agents in their stable states, and a technique to do so has been desired.

As a previous method for solving the problems described above, there are techniques to fix flavoring agents. For example, the proposed methods include synthetic clove oil placed in microcapsules (U.S. Patent No. 3,623,489) and menthol placed in microcapsules (U.S. Patent 3,550,598). Furthermore, other known methods include a medium or carrier impregnated or allowed to adsorb flavoring agents, such as zeolite impregnated with menthol and added to cigarette filters (Japanese Kokai Patent Application No. Sho 59[1984]-17974), water-containing metal oxide or chelate compound allowed to absorb flavoring agents (Japanese Kokoku Patent No. Sho 57[1982]-22314), etc. However, the effects of retaining the aromas of volatile flavoring agents by using those known methods are not necessarily satisfactory, or the materials used to

retain flavoring agents give off an odor or burning odor undesirable for smoking, causing some restrictions on their applications.

The present inventors achieved this invention for the purpose of providing a smoking material making it possible to retain the flavor and aroma of smoking products after they are prepared and give uniform aroma and taste from the start to the end of smoking.

#### Means to solve the problems

The inventors of this invention found that carboxymethylcellulose (abbreviated CMC, below), which is used as a binder of various raw materials in the present process for the production of reconstituted tobacco sheets, has an action of covering the surface of a carrier substance impregnated with a volatile flavoring agent by adsorption and effectively preventing evaporation of the flavoring agent, and they arrived at this invention. Specifically, this invention is a smoking composition sheet, characterized by compounding a reconstituted tobacco raw material sheet containing carboxymethylcellulose as a binder impregnated with a menthol solution by adsorption to form a slurry which is subsequently molded and dried to enhance the volatile flavoring agent-retaining ability. The reconstituted tobacco sheet in this case is a smoking composition reconstituted by suitably selecting powder tobacco obtained by pulverizing leaf and leaf vein scraps generated by processing tobacco leaves, fiber such as pulp, inorganic additives such as limestone, perlite, etc., moisture retainers comprising glycerol, glycol and other polyols and other smoking composition components. Furthermore, the volatile flavoring agent is selected from those flavoring agents which are conventionally used as a constituting component in the production of tobacco products and have volatility such as menthol, cinnamic aldehyde, eugenol, linalool, [illegible] acetate, citronellol, nerol, anisole, geraniol, geranyl aldehyde, cinnamic aldehyde [sic], etc. As a powder or granular adsorbent usable as a flavoring agent carrier in this invention, the use of activated carbon is optimal because its retaining power is large, and there are no adverse effects on the flavor and aroma of smoking.

The process for the production of the smoking composition sheet of this invention is explained as follows. Activated carbon as a flavoring agent adsorbent is pulverized to 100 mesh or finer, and the powder prepared is used as it is or after drying at about 200°C for about 2 h to remove its moisture content. The most volatile flavoring agent, menthol, is selected from those flavoring agents used for tobacco products, the activated carbon powder prepared as described above is soaked in an ethanol solution of a required amount of the dissolved flavoring agent, the mixture is stirred and allowed to stand to impregnate the activated carbon with the flavoring agent sufficiently by soaking and adsorption. Incidentally, the soaking time may be about 10-20 min. The flavoring agent-containing activated carbon prepared is added to reconstituted tobacco raw materials in the amount to obtain a required amount of flavoring agent, and

subsequently the known process for the production of reconstituted tobacco is used to carry out work units of mixing, molding and drying successively to obtain a smoking composition sheet.

The smoking composition sheet of this invention is compounded with the raw material leaves or intermediate cuts for cigarettes, pipe tobaccos, etc., in a prescribed amount to obtain tobacco products.

#### Application Example 1

Commercially available coconut shell activated carbon was pulverized to 100 mesh or finer to obtain a micropowder which was subsequently dried in an isothermal electric dryer at 200°C for 2 h. On the other hand, 35 mL ethanol were placed in a suitable container, 3 g menthol were dissolved first, then 10 g of the activated carbon were added while stirring, the mixture was allowed to stand at a room temperature for 10 min, and the activated carbon was impregnated sufficiently with the menthol solution. Separately, 85 g of a reconstituted tobacco raw material mixture were prepared from 8 g of powder of leaves of yellow tobacco species, 28 g limestone powder, 23 g ammonium salt of CMC, 11 g pulp and 15 g of a powder of squeezed orange residue. 85 g of the mixture and all of the menthol-adsorbed activated carbon were placed in a suitable container, 350 mL water and 12 g glycerol were added, and the mixture was stirred thoroughly to obtain a slurry. The slurry prepared was dried and molded in an experimental small slurry-molding dryer to prepare about 100 g of a smoking composition sheet of 0.15-mm thickness and 82 g/m<sup>2</sup> unit weight.

To prepare a comparative control product to the smoking composition sheet prepared as described above, a menthol-adsorbed smoking composition sheet containing no activated carbon (called control product, below) was prepared. Specifically, 35 mL methanol were placed in a suitable container, and 3 g menthol were dissolved. Separately, 85 g of a reconstituted tobacco raw material mixture were prepared from 8 g of powder of leaves of yellow tobacco species, 28 g limestone powder, 23 g ammonium salt of CMC, 11 g pulp and 15 g of a powder of squeezed orange residue. 85 g of the mixture and all of the menthol-solution were placed in a suitable container, 350 mL water and 12 g glycerol were added, and the mixture was stirred thoroughly to obtain a slurry. The prepared slurry was dried and molded in an experimental small slurry-molding dryer to prepare about 90 g of a smoking composition sheet of 0.15-mm thickness and 80 g/m<sup>2</sup> unit weight.

#### Application Example 2

The smoking composition sheet and control product prepared in Application Example 1 were allowed to stand open in a room at a temperature of 22°C and relative humidity of 60%, and the amount of menthol retained by the smoking composition and control product was determined

at a constant interval by using gas chromatography. The menthol quantitative determination method used was as follows. A 200-mL flask with a ground stopper was charged with 5 g of the smoking composition sheet (sample), 25 mL 0.5N aqueous NaOH solution were added, and the mixture was shaken vigorously to dissolve the ammonium salt of CMC and obtain a sample in a powder form. Subsequently, 100 mL CH<sub>2</sub>Cl<sub>2</sub> were added, the mixture was shaken at 30°C for about 15 h to dissolve the menthol in the sample and transfer it into CH<sub>2</sub>Cl<sub>2</sub>. The CH<sub>2</sub>Cl<sub>2</sub> layer was isolated and analyzed by gas chromatography. The conditions used were as follows.

- a. Instrument: Hitachi, Model 163
- b. Column: butane-1,4-diol adipate 10% Chromosorb (AW 60-80 mesh) glass column 2 m
- c. Temperature, column: 160°C, injection port: 230°C
- d. Detector: FID
- e. Carrier gas: N<sub>2</sub>, 60 mL/min.

Table 1 shows the results of analysis. As apparent from the results shown in Table 1, the amount of menthol retained was drastically reduced during the period from immediately after production to about 1 month in the control product, only a small amount was found to be retained after 1 month, and no menthol was found to be retained subsequently. On the other hand, in the smoking composition sheet with activated carbon added, the amount of menthol retained was reduced markedly to a certain extent during the first month after production, but subsequently, it showed only a gradual decrease, and even after 24 months, there was still menthol in an amount sufficient to provide a cool and refreshing taste.

Table 1

① 試験物の種類	② シート状喫煙用組成物 1 g 中のメントール量								④
	③ 製造直後	1ヶ月後	2ヶ月後	3ヶ月後	4ヶ月後	5ヶ月後	6ヶ月後	24ヶ月後	
⑤ シート状喫煙用組成物	2.6 mg	1.3 mg	1.3 mg	1.1 mg	1.1 mg	1.1 mg	1.1 mg	7 mg	
⑥ 対照品	2.8 mg	1 mg	0	0	0	0	0	0	

- Key:
- 1 Sample
  - 2 Amount of menthol in 1 g of smoking composition sheet
  - 3 Immediately after production
  - 4 After \_\_\_\_ months
  - 5 Smoking composition sheet

- 6 Control product
- 7 Less than 1 mg

#### Application Example 3

The smoking composition sheet and control product prepared in Application Example 1 were respectively cut, and 10 wt% was added to a tobacco cut material to produce a pipe tobacco product called Rock'n Chair (trade name of Nippon Tobacco) to obtain pipe tobacco products which were smoked subsequently. As a result, the cool and refreshing taste of the menthol was experienced in a uniform and stable state from the beginning to the end of smoking. On the other hand, in the pipe tobacco with the control product compounded, the cool and refreshing taste of menthol was experienced strongly at the beginning of pipe smoking, but it was gradually reduced, and there was hardly any near the end of smoking.

#### Application Example 4

The same procedures as those used in Application Example 1, except that the amount of menthol used was reduced to  $\frac{1}{2}$ , were carried out to prepare a smoking composition sheet of this invention and control product. The smoking composition sheet of this invention and control product were respectively cut, cigarettes were prepared by rolling them, stored in an open state in a room at a temperature of 22°C and relative humidity of 60%, and the extent of the cool and refreshing taste of the menthol retained was investigated at 1-month intervals for 24 months by smoking the cigarettes. As a result, the cool and refreshing taste of the menthol was detected in cigarettes containing the smoking composition sheet of this invention even after 24 months. However, the cool and refreshing taste of the menthol was hardly detected after only a month in cigarettes containing the control product.

#### Effect of the invention

In the smoking composition sheet of this invention, strongly volatile menthol is retained by adsorption on activated carbon powder granules, and at the same time, the surface of the

..... is covered with CMC. Consequently, the menthol in the smoking